



## DECLARATION

I, Yoshiaki TODAKA of c/o The Patent Corporate Body ARUGA PATENT OFFICE, 3-6, Nihonbashiningyocho 1-chome, Chuo-ku, Tokyo 103-0013 Japan do solemnly and sincerely declare that I well understand both Japanese and English languages and that I believe the attached English version is a true and complete translation of the Japanese Patent Application No. 2002-269185 filed on September 13, 2002 in the name of Kao Corporation.

December 11, 2006

A handwritten signature in cursive script, appearing to read "Yoshiaki TODAKA", written over a horizontal line.

Yoshiaki TODAKA



Docket No.: 242826US0



ATTORNEYS AT LAW

COMMISSIONER FOR PATENTS  
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RE: Application Serial No.: 10/660,722  
Applicants: Tsutomu NISHIDE, et al.  
Filing Date: September 12, 2003  
For: OIL OR FAT COMPOSITIONS  
Group Art Unit: 1761  
Examiner: PADEN, C.A.

RESPONSE UNDER 37 CFR 1.116-  
EXPEDITED PROCEDURE EXAMINING  
GROUP 1761 "

SIR:

Attached hereto for filing are the following papers:

**RESPONSE AND REQUEST FOR RECONSIDERATION UNDER 37 C.F.R. 1.116/  
CERTIFIED ENGLISH TRANSLATIONS OF (3) JAPANESE PRIORITY DOCUMENTS-  
JP 2002-269185; JP 2002-275805; and JP 2002-313597**

Our check in the amount of \$ -0- is attached covering any required fees. In the event any variance exists between the amount enclosed and the Patent Office charges for filing the above-noted documents, including any fees required under 37 C.F.R. 1.136 for any necessary Extension of Time to make the filing of the attached documents timely, please charge or credit the difference to our Deposit Account No. 15-0030. Further, if these papers are not considered timely filed, then a petition is hereby made under 37 C.F.R. 1.136 for the necessary extension of time. A duplicate copy of this sheet is enclosed.

Respectfully submitted,

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[Document Name] SPECIFICATION

[Title of the Invention] OIL or FAT COMPOSITIONS

[Claims]

[Claim 1] An oil or fat composition comprising the following ingredients (A), (B), (C) and (D):

(A) 100 weight parts of an oil or fat containing 60 to 100 wt% of diglycerides in which a content of unsaturated fatty acids in constituent fatty acids is from 80 to 100 wt%;

(B) 0.001 to 1 weight parts of a carboxylic acid selected from C<sub>2-8</sub> hydroxycarboxylic acids, dicarboxylic acids and tricarboxylic acids, and salts and derivatives thereof;

(C) 0.001 to 5 weight parts of an antioxidant; and

(D) 0.05 to 4.7 weight parts of a plant sterol.

[Claim 2] An oil or fat composition according to claim 1, wherein said constituent fatty acids in said ingredient (A) have a fatty acid composition comprising 20 to 65 wt% of oleic acid, 15 to 65 wt% of linoleic acid, less than 15 wt% of linolenic acid, and not greater than 10 wt% of saturated fatty acids.

[Claim 3] An oil or fat composition according to claim 1 or 2, further comprising (E) 0.01 to 2.5 weight parts of a crystallization inhibitor.

[Claim 4] A food comprising an oil or fat composition according to any one of claims 1-3.

[Detailed Description of the Invention]

[0001]

### [Field of the Invention]

This invention relates to oil or fat compositions, each of which is high in the content of diglycerides having excellent health-promoting functions and even in cold temperature areas or high-humidity areas, is prohibited in the occurrence of solidification or turbidity and is good in work efficiency during cooking and also in the flavor and texture of cooked foods.

[0002]

### [Conventional Art]

Diglycerides have been found to be effective for improving blood cholesterol levels (see Patent Document 1) and also to be effective for curtailing the accumulation of body fat and preventing adiposis (see Patent Documents 2 and 3). These effects are considered to be attributed to the inhibition of an increase in the level of fat in postcibal blood. Further, the use of diglycerides in cooking oil is known to bring about such merits that upon frying, foaming is reduced and an improved flavor and texture are assured (see Patent Documents 4 to 6). In addition, diglycerides are also indicated to be applicable to emulsified foods (see Patent Documents 7 to 9). With the foregoing in view, oil or fat compositions containing diglycerides at high contents have found wide spread utility as edible oils or fats for home use.

In the meantime, a technique has been disclosed in which an antioxidant and a crystallization inhibitor are added to

diglycerides to improve their storage stability (see Patent Document 3). Another method has also been disclosed, in which an organic acid is added to inhibit disproportionation in a deodorization step (see Patent Document 10).

[0003]

[Patent Document 1]

The pamphlet of PCT International Publication  
No. WO 99/48378

[Patent Document 2]

JP-A-04-300826

[Patent Document 3]

JP-A-10-176181

[Patent Document 4]

JP-A-02-190146

[Patent Document 5]

JP-A-07-016051

[Patent Document 6]

JP-A-09-154494

[Patent Document 7]

JP-B-04-034367

[Patent Document 8]

US-B-5,879,735

[Patent Document 9]

JP-A-03-008431

[Patent Document 10]

JP-A-04-261497

[0004]

## [Problems to Be Solved by the Invention]

As is appreciated from the foregoing, there is an increasing desire not only in Japan but also in various countries in the world toward the use of diglycerides for their superb health-promoting functions.

Depending on use conditions or storage conditions, however, the external appearances of diglycerides-containing oils or fats may be impaired in some instances, because compared with triglycerides, diglycerides tend to become solid in cold temperature areas or tend to become turbid in high-humidity areas. Further, use of an oil or fat with moisture absorbed therein leads stronger emission of an unpleasant smell during cooking so that cooked foods may be impaired in flavor and texture in some instances.

An object of the present invention is, therefore, to provide an oil or fat composition, which is high in the content of diglycerides having excellent health-promoting functions and even in cold temperature areas or high-humidity areas, is prohibited in solidification and the occurrence of turbidity, and is good in work efficiency during cooking and also in the flavor and texture of cooked foods.

[0005]

## [Means for Solving the Problems]

The present inventors investigated to determine causes of the occurrence of turbidity or solidification in diglycerides-containing oils or fats. Concerning solidification in cold temperature areas, diglycerides are considered to be susceptible to solidification through hydrogen bonding between hydroxyl groups because they contain one more hydroxyl group in a molecule than conventional oils or fats (triglycerides). As to turbidity in high-humidity areas, on the other hand, the high compatibility of the hydroxyl groups of diglycerides with water is considered to make an oil or fat more hygroscopic to produce turbidity. Such solidification and turbidity are unique phenomena caused by the higher polarity of diglycerides than triglycerides. It has been found that control of the content of plant sterols in a diglycerides-containing oil or fat by addition of a specific organic acid and an antioxidant makes it possible to pronouncedly inhibit such turbidity and solidification, to markedly prevent occurrence of an unpleasant odor while cooking, and further, to significantly improve work efficiency.

[0006]

The present invention, therefore, provides an oil or fat composition comprising the following ingredients (A), (B), (C) and (D):

(A) 100 weight parts of an oil or fat containing 60 to 100 wt% of diglycerides in which a content of unsaturated fatty

acids in constituent fatty acids is from 80 to 100 wt%;

(B) 0.001 to 1 weight parts of a carboxylic acid selected from C<sub>2-8</sub> hydroxycarboxylic acids, dicarboxylic acids and tricarboxylic acids, and salts and derivatives thereof;

(C) 0.001 to 5 weight parts of an antioxidant; and

(D) 0.05 to 4.7 weight parts of a plant sterol.

[0007]

[Modes for Carrying Out the Invention]

The present invention employs as an oil or fat composition an oil or fat which contains diglycerides in a proportion of from 60 to 100 wt% (hereinafter indicated merely by "%"). Oils or fats containing diglycerides at a high content are not used especially in high-humidity areas although they are used in Japan as described above. Conventionally, it has not been known at all that turbidity quickly occurs under such conditions.

[0008]

The oil or fat useful as the ingredient (A) in the oil or fat composition according to the present invention contains diglycerides in a proportion of from 60 to 100%. From the standpoint of physiological effects, industrial productivity and external appearance, however, it is preferred to contain them in a proportion of from 70 to 100%, more preferably from 80 to 97%, even more preferably from 80 to 95%.

[0009]

Unsaturated fatty acids may amount to from 80 to 100% of



the constituent fatty acids of the diglycerides. From the standpoint of external appearance and physiological effects, however, C<sub>10-24</sub>, preferably C<sub>16-22</sub> unsaturated fatty acids may preferably amount to from 90 to 100%, with from 93 to 98% being more preferred and from 94 to 98% being even more preferred.

[0010]

Described specifically, the content of oleic acid in the fatty acids making up the diglycerides may range from 20 to 65%, preferably from 25 to 60%, more preferably from 30 to 50%, even more preferably from 30 to 45% from the standpoint of external appearance and balanced ingestion of fatty acids. From the standpoint of physiological effects, the content of olein-olein diglyceride may preferably be lower than 45%, with from 0 to 40% being particularly preferred.

[0011]

The content of linoleic acid in the fatty acids making up the diglycerides may range from 15 to 65%, preferably from 20 to 60%, more preferably from 30 to 55%, even more preferably from 35 to 50% from the standpoint of external appearance and balanced ingestion of fatty acids. Further, from the standpoint of oxidation stability and physiological effects, the content weight ratio of linoleic acid/oleic acid may range from 0.01 to 2.0, preferably from 0.1 to 1.8, even more preferably from 0.3 to 1.7.

[0012]

The content of linolenic acid in the fatty acids making up the diglycerides may be lower than 15%, preferably from 0 to 13%, more preferably from 1 to 10%, even more preferably from 2 to 9% from the standpoint of external appearance, balanced ingestion of fatty acids and oxidation stability. Linolenic acid is known to include  $\alpha$ -linolenic acid and  $\gamma$ -linolenic acid as isomers, with  $\alpha$ -linolenic acid being preferred.

[0013]

Among the fatty acids making up the diglycerides, the content of fatty acids may be 20% or lower, preferably from 0 to 15%, more preferably from 0 to 10%, even more preferably from 2 to 7%, still even more preferably from 2 to 6% from the standpoint of external appearance, physiological effects and industrial productivity. Among the fatty acids,  $C_{14-24}$ , especially  $C_{16-22}$  fatty acids are preferred, with palmitic acid and stearic acid being more preferred.

[0014]

The content of trans acids in the fatty acids making up the diglycerides may desirably range from 0 to 10%, preferably from 0.1 to 5% from the standpoint of physiological effects, external appearance and industrial productivity. The remaining constituent fatty acids may preferably be  $C_{14-24}$ , more preferably  $C_{16-22}$  fatty acids.

[0015]

From the standpoint of physiological effects, storability,

industrial productivity and flavor, the proportion of 1,3-diglycerides in the diglycerides may be preferably 50% or higher, more preferably from 60 to 100%, still more preferably from 65 to 90%, even more preferably from 65 to 80%.

[0016]

The oil or fat useful as the ingredient (A) in the present invention may contain triglycerides in a proportion of preferably from 0 to 40%, more preferably from 0 to 30%, still more preferably from 2.9 to 20%, even more preferably from 4.9 to 20% from the standpoint of physiological effects, industrial productivity and external appearance.

[0017]

It is preferred from the standpoint of physiological effects and industrial productivity that  $C_{10-24}$ , preferably  $C_{16-22}$  unsaturated fatty acids amount to from 80 to 100%, preferably from 90 to 100%, more preferably from 93 to 100%, still more preferably from 93 to 98%, even more preferably 94 to 98% of the constituent fatty acids of the triglycerides.

[0018]

In the oil or fat useful as the ingredient (A) in the present invention, the content of monoglycerides may desirably range from 0 to 10%, preferably from 0.1 to 5%, more preferably from 0.1 to 1.5%, still more preferably from 0.1 to 1.3%, even more preferably from 0.1 to 1% from the standpoint of flavor, external appearance, emulsification, prevention of smoking, industrial

productivity and the like. It is preferred from the standpoint of industrial productivity that the constituent fatty acids of the monoglycerides are the same as those of the diglycerides.

[0019]

The content of free fatty acids (and salts thereof) in the oil or fat useful as the ingredient (A) in the present invention may desirably be lowered to 3.5% or less, and may range preferably from 0 to 2%, more preferably from 0 to 1%, still more preferably from 0 to 0.5%, even more preferably from 0.05 to 0.2% from the standpoint of flavor, prevention of smoking, and industrial productivity.

[0020]

In the whole fatty acids making up the oil or fat useful as the ingredient (A) in the present invention, the content of fatty acids containing 4 or more carbon-carbon double bonds may range from 0 to 40%, preferably from 0 to 20%, more preferably from 0 to 10%, even more preferably from 0 to 1%, with an oil or fat substantially free of fatty acids containing 4 or more carbon-carbon double bonds being more preferred, from the standpoint of oxidation stability, work efficiency, physiological effects, coloration and the like.

[0021]

A source of the oil or fat useful as the ingredient (A) in the present invention can be either a vegetable or animal oil or fat. Specific sources can include rapeseed oil, sunflower

oil, corn oil, soybean oil, rice oil, safflower oil, cottonseed oil, and beef tallow. These oils and fats can also be used as sources after adjusting their fatty acid compositions by fractionation, blending, hydrogenation, ester interchange or the like.

[0022]

The oil or fat useful as the ingredient (A) in the present invention can be obtained by an esterifying reaction between fatty acids derived from the above-mentioned oil or fat and glycerin, or an ester interchange reaction between such an oil or fat and glycerin, or a like reaction. Excess monoglycerides formed by the reaction can be eliminated by molecular distillation or chromatography. It is preferred to conduct these reactions with 1,3-selective lipase or the like under enzymatically mild conditions as this procedure is superior in flavor and the like, although the reactions can also be conducted as chemical reactions by making use of an alkali catalyst or the like.

[0023]

The oil or fat composition according to the present invention is required to contain, as the ingredient (B), a carboxylic acid selected from hydroxycarboxylic acids, dicarboxylic acids and tricarboxylic acids, and salts and derivatives thereof. The content of the carboxylic acid ranges from 0.001 to 1 weight parts per 100 weight parts of the

ingredient(A). From the standpoint of low-temperature stability, high-humidity stability, external appearance and oxidation stability, however, the content of the carboxylic acid may range preferably from 0.0012 to 0.7 weight parts, more preferably from 0.0015 to 0.5 weight parts, even more preferably from 0.0025 to 0.3 weight parts. The carbon number of the carboxylic acid is required to be from 2 to 8, with 2 to 6 being preferred and 4 to 6 being more preferred. Preferred specific examples can include citric acid, succinic acid, maleic acid, oxalic acid, aconitic acid, itaconic acid, citraconic acid, tartaric acid, fumaric acid and malic acid, with citric acid, tartaric acid and malic acid being more preferred.

[0024]

Examples of the above-described derivatives of  $C_{2-8}$  hydroxycarboxylic acids, dicarboxylic acids and tricarboxylic acids can include glyceride derivatives such as monoglycerides citrates, diglycerides citrates, monoglycerides succinates and diglycerides succinates. Specifically, monoglyceride citrate (crystal) [product of ADM (Archer Daniels Midland Co.)], purified crystalline monoglyceride citrate (product of Fuso Chemical Co., Ltd.) and "Step SS" (product of Kao Corporation) can be exemplified as commercial products. Illustrative salts of the above-mentioned carboxylic acids can include their alkali metals and alkaline earth metals, with the sodium salts and calcium salts being preferred.

## [0025]

As carboxylic acids, extracts and crude drugs with such carboxylic acids contained therein can also be used. Usable examples of the extracts and crude drugs can include products commercially available in the form of powders, concentrates or the like produced by extraction from fruits such as lemons, yuzu (Japanese citrons) and ume (Japanese apricots). These extracts and crude drugs can each be added in such an amount that the content of the carboxylic acid contained therein falls within the above-described range.

## [0026]

The content of the C<sub>2-8</sub> hydroxycarboxylic acid, dicarboxylic acid or tricarboxylic acid or the salt thereof may range desirably from 0.001 to 0.01 weight parts, more desirably from 0.0012 to 0.007 weight parts, still more desirably from 0.0015 to 0.0045 weight parts, even more desirably from 0.0025 to 0.0034 weight parts per 100 weight parts of the ingredient (A) from the standpoint of low-temperature stability, high-humidity stability, external appearance, oxidation stability, work efficiency and flavor.

## [0027]

The content of the derivative of the C<sub>2-8</sub> hydroxycarboxylic acid, dicarboxylic acid or tricarboxylic acid may range preferably from 0.01 to 1 weight parts, more preferably from 0.05 to 0.7 weight parts, still more preferably from 0.1 to 0.5

weight parts, even more preferably from 0.15 to 0.3 weight parts per 100 weight parts of the ingredient (A) from the standpoint of low-temperature stability, high-humidity stability, external appearance, oxidation stability, work efficiency, flavor and texture.

[0028]

Combined use of the  $C_{2-8}$  hydroxycarboxylic acid, dicarboxylic acid or tricarboxylic acid or the salt thereof with the derivative thereof is preferred especially from the standpoint of texture, external appearance, oxidation stability and flavor.

[0029]

In the present invention, the content of the ingredient (B) in the oil or fat composition can be measured by HPLC, colorimetry making use of ortho nitrophenylhydrazine, or the like. For example, measurement of citric acid by colorimetry can be carried out in the following manner.

An oil or fat (20 g), which has been heated to 60°C, is placed in a 100-mL separating funnel, and subsequent to the addition of warm water of 60°C (5 mL), the contents are vigorously shaken for 2 minutes. The resulting mixture is then allowed to stand until it separates into layers, and the lower layer is collected as a sample solution. This sample solution (2 mL), an ONPH solution<sup>1)</sup> (1 mL) and an ETC solution<sup>2)</sup> (1 mL) are placed in a 10-mL graduated flask. After the graduated flask is tightly



sealed, its contents are heated at 40°C for 30 minutes. A 1.5 mol/L solution of sodium hydroxide (1 mL) is then added, followed by heating at 60°C for 15 minutes. After the mixture is allowed to cool down to room temperature, an absorption at 540 nm is measured. From a calibration line prepared using aqueous citric acid solutions the concentrations of which were known, the content of citric acid is determined in accordance with the following formula:

[0030]

The content of citric acid in the oil or fat  
= the amount of citric acid determined from the  
calibration line ÷ 4

[0031]

1) ONPH solution: a solution of orthonitrophenyl-hydrazine hydrochloride (ONPH) (53.6 mg) in 0.2 mol/L hydrochloric acid (10 mL).

2) ETC solution: a solution of 1-ethyl-3-(3-dimethylaminopropyl)-carbodiimide hydrochloride (ETC) (287.6 mg) in a 6% aqueous solution (10 mL) of pyridine.

[0032]

The oil or fat composition according to the present invention is also required to contain the antioxidant as the ingredient (C). The antioxidant, in combination with the ingredient (B), exhibits synergistic effects, and serves not only to significantly improve work efficiency upon cooking but

also to pronouncedly lessen the turbidity and solidification.

[0033]

From the standpoint of work efficiency, stability and coloration, the content of the antioxidant as the ingredient (C) ranges from 0.001 to 5 weight parts per 100 weight parts of the ingredient (A), but may range preferably from 0.004 to 0.5 weight parts, more preferably from 0.004 to 0.4 weight parts, even more preferably from 0.008 to 0.3 weight parts.

In general, the antioxidant as the ingredient (C) can preferably be one useful in foods. Illustrative are vitamin E, buthylhydroxytoluene (BHT), butylhydroxyanisole (BHA), tert-butylhydroquinone (TBHQ), vitamin C and derivatives thereof, phospholipids, and natural antioxidants such as rosemary extract, among which vitamin E, vitamin C and derivatives thereof, and rosemary extract are preferred. It is more preferred to use two or more of these antioxidants in combination.

[0034]

More specifically,  $\alpha$ ,  $\beta$ ,  $\gamma$  or  $\delta$ -tocopherol or a mixture thereof can be used as vitamin E. From the viewpoint of oxidation stability,  $\delta$ -tocopherol is particularly preferred. Commercial products of vitamin E can include "E-MIX D" and "E-MIX 80" (products of Eisai Co., Ltd.), "MDE-6000" (product of Yashiro Co., Ltd.), and "E-Oil 400" (product of Riken Vitamin Co., Ltd.).

In the present invention, the content of vitamin E may

range preferably from 0.01 to 0.4 weight parts, more preferably from 0.02 to 0.3 weight parts, most preferably from 0.05 to 0.2 weight parts, all in terms of tocopherol per 100 weight parts of the ingredient (A).

[0035]

As vitamin C and derivatives thereof, those soluble in diglycerides-containing oils or fats are preferred. More preferred can be higher fatty acid esters, for example, those having C<sub>12-22</sub> acyl groups. Still more preferred are L-ascorbic acid palmitate and L-ascorbic acid stearate, with L-ascorbic acid palmitate being even more preferred.

In the present invention, the content of vitamin C or the derivative thereof may range preferably from 0.004 to 0.1 weight parts, more preferably from 0.006 to 0.08 weight parts, even more preferably from 0.008 to 0.06 weight parts, all in terms of ascorbic acid per 100 weight parts of the ingredient (A).

[0036]

The oil or fat composition according to the present invention is also required to contain the plant sterol as the ingredient (D). The plant sterol is an ingredient having a cholesterol-lowering effect and, when used in combination with the ingredients (B) and (C), improves the efficiency of cooking work, low-temperature stability and high-humidity stability of the diglycerides-containing oil or fat. In the present invention, the content of the plant sterol is from 0.05 to 4.7

weight parts per 100 weight parts of the ingredient (A), with 0.3 to 4.7 weight parts being preferred. In a commercial oil or fat composition produced using as a raw material fatty acids obtained by distillation, the content of a plant sterol may have been lowered in general. In such a case, a plant sterol can be added in such an amount that its content falls within a range of from 0.05 to 4.7 weight parts.

Examples of the plant sterol can include free sterols such as  $\alpha$ -sitosterol,  $\beta$ -sitosterol, stigmasterol, campesterol,  $\alpha$ -sitostanol,  $\beta$ -sitostanol, stigmastanol, campestanol and cycloartenols, and their esters such as their fatty acid esters, ferulate esters and cinnamate esters.

[0037]

To the oil or fat composition according to the present invention, the crystallization stabilizer can preferably be added further as the ingredient (E) to improve its low-temperature stability and high-humidity stability.

Examples of the crystallization stabilizer can include polyol fatty acid esters such as polyglyceryl ricinoleate, polyglyceryl fatty acid esters, sucrose fatty acid esters, sorbitan fatty acid esters, polyoxyethylene sorbitan fatty acid esters, and propylene glycol fatty acid esters. Preferred examples of the polyol fatty acid esters can include polyglyceryl fatty acid esters, sucrose fatty acid esters and sorbitan fatty acid esters having HLB values not greater than 4, more preferably of from

0.1 to 3 [as measured by the Griffin's calculation formula reported in J. Soc. Cosmet. Chem., 1, 311 (1949)].

In the present invention, it is preferred from the standpoint of improving the stability at low temperatures to contain the crystallization inhibitor in a proportion of from 0.01 to 2.5 weight parts, more preferably from 0.02 to 0.5 weight parts, even more preferably from 0.05 to 0.2 weight parts per 100 weight parts of the ingredient (A).

[0038]

Preferably, the edible oil or fat according to the present invention may additionally contain a silicone from the standpoint of low-temperature stability, defoamability, oxidation stability and flavor. As the silicone, a preparation ("KS-66", "KS-69", "KF-96", "KM-72" or the like, product of Shin-Etsu Chemical Co., Ltd.; or "THF450", "TSA737" or the like, product of Toshiba Silicone Co., Ltd.) known as a defoaming agent for addition to foods such as dimethylpolysiloxane can be mentioned.

In the present invention, the silicone may be contained in a proportion of preferably 0.0003 weight parts or greater, more preferably from 0.0005 to 0.001 weight parts, even more preferably from 0.001 to 0.006 weight parts per 100 weight parts of the ingredient (A) from the standpoint of improvements in low-temperature stability.

[0039]

The edible oil or fat composition according to the present

invention can be obtained by adding the above-described ingredients (B) to (D) to the diglycerides-containing oil or fat of the above-described composition, adding a silicone or the like as needed, and heating and stirring the resultant mixture as needed. Rosemary extract, the vitamin C derivative, vitamin E and the like can be added after dissolving them in a solvent beforehand.

[0040]

The oil or fat composition obtained as described above can be applied as an edible oil or fat to various foods, because it is good in external appearance, work efficiency, flavor, texture and the like.

As a food, it can be used in oil or fat-containing processed foods which contains the oil or fat composition as parts of the foods. Examples of such oil or fat-containing processed foods can include health-promoting foods, functional foods and specific health foods which exhibit specific functions to achieve the promotion of health. Specific products can include bakery foods such as breads, cakes, biscuits, pies, pizza crusts, and bakery mixes; oil-in-water (o/w) emulsions such as soups, sauces, dressings, mayonnaises, coffee creams, ice creams, and whipped creams; water-in-oil (w/o) emulsions such as margarines, spreads, and butter creams; snacks such as potato chips; confectioneries such as chocolates, caramels, candies, and desserts; processed meat foods such as hams, sausages, and hamburger steaks; milk

products such as milks, cheeses, and yogurts; doughs; enrober oils or fats; filling oils or fats; noodles; frozen foods; pouch-packaged foods; drinks; and roux. These oil or fat-containing processed foods can each be produced by adding, in addition to the above-described oil or fat, food materials which are commonly employed in the oil or fat-containing processed food. The oil or fat composition according to the present invention can be added generally in a proportion of from 0.1 to 100% to a food, with from 1 to 80% being more preferred, although its proportions varies depending on the kind of food.

The oil or fat composition according to the present invention can also be used as a food material such as a cooking oil useful for deep-fried foods and pan-fried foods. It is particularly suited for cooking or otherwise preparing delicatessens such as croquettes, *tempura* (deep-fried fish and vegetables), fried pork cutlets, *kara-age* (foods fried without breading), fried fish, and egg rolls; snacks such as potato chips, tortilla chips, and fabricated potatoes; fried confectioneries such as fried rice crackers; fried potatoes; fried chicken; donuts; instant noodles; and the like.

[0041]

When an oil or fat derived from a food material is contained because of the formula of a mix or the like, the weight ratio of the oil or fat derived from the food material to the oil or fat composition according to the present invention may range

preferably from 95:5 to 1:99, more preferably from 95:5 to 5:95, still more preferably from 85:15 to 5:95, especially from 40:60 to 5:95.

The oil or fat composition according to the present invention can be used in o/w emulsions. The weight ratio of the oil phase to the water phase may range from 1:99 to 90:10, preferably from 10:90 to 80:20, more preferably from 30:70 to 75:25. An emulsifier may be contained preferably in a proportion of from 0.01 to 5%, with 0.05 to 3% being particularly preferred. Examples of the emulsifier can include various proteins such as egg proteins, soybean proteins, milk proteins, proteins isolated from these proteins, and (partial) hydrolysates of these proteins; sucrose fatty acid esters; sorbitan fatty acid esters; polyoxyethylene sorbitan fatty acid esters; monoglyceryl fatty acid esters; polyglyceryl fatty acid esters; polyglyceryl ricinoleate; glyceryl organic acid esters; propylene glycol fatty acid esters; and lecithin and enzymatic hydrolysates thereof. A stabilizer may be contained preferably in a proportion of from 0 to 5%, with from 0.01 to 2% being particularly preferred. Examples of the stabilizer can include thickening polysaccharides and starches, such as xanthan gum, gellan gum, guar gum, carageenan, pectin, tragacanth gum, and glucomannan (*konjak* mannan). It is also possible to use one or more of taste-imparting substances such as salt, sugar, vinegar, fruit juices, and seasonings; fragrance additives such as spices and



flavors; color additives, preservatives; antioxidants; and the like. Using these materials, oil or fat-containing o/w foods such as mayonnaises, dressings, coffee creams, ice creams, whipped creams and drinks can be prepared by conventional procedures.

[0042]

The oil or fat composition according to the present invention can also be used in w/o emulsions. The weight ratio of the water phase to the oil phase may range from 85:15 to 1:99, preferably from 80:20 to 10:90, more preferably from 70:30 to 35:65. An emulsifier may be contained preferably in a proportion of from 0.01 to 5%, with 0.05 to 3% being more preferred. Examples of the emulsifier can include various proteins such as egg proteins, soybean proteins, milk proteins, proteins isolated from these proteins, and (partial) hydrolysates of these proteins; sucrose fatty acid esters; sorbitan fatty acid esters; polyoxyethylene sorbitan fatty acid esters; monoglyceryl fatty acid esters; polyglyceryl fatty acid esters; polyglyceryl ricinoleate; glyceryl organic acid esters; propylene glycol fatty acid esters; and lecithin and enzymatic hydrolysates thereof. It is also possible to use one or more of taste-imparting substances such as salt, sugar, vinegar, fruit juices, and seasonings; fragrance additives such as spices and flavors; stabilizers such as thickening polysaccharides and starches; color additives, preservatives; antioxidants; and the

like. Using these materials, oil or fat-containing w/o foods such as margarines, spreads, and butter creams can be prepared by conventional procedure.

[0043]

[Examples]

Example 1

(1) Production of oils

The following oils were produced.

[0044]

Oil A

Soybean fatty acids with the content of saturated fatty acids lowered by wintering (455 weight parts), rapeseed oil fatty acids (195 weight parts) and glycerin (107 weight parts) were subjected to esterification at 0.07 hPa and 40°C for 5 hours by using "Lipozyme IM" (product of Novo Nordisk Bioindustries, Ltd.). Subsequently, the enzyme was filtered off, molecular distillation was conducted at 235°C, and further, bleaching and water washing were carried out. To 150 weight parts of the oil, a 10% aqueous solution of citric acid (7.5 weight parts) was then added. Subsequent to stirring at 60°C for 20 minutes, dehydration was conducted at 110°C and 0.27 hPa. The oil was then deodorized at 235°C for 2 hours to produce Oil A.

Oil B

Rapeseed fatty acids (650 weight parts) and glycerin (107 weight parts) were subjected to similar esterification and

post-treatments as in Oil A to produce Oil B.

### Oil C

Fatty acids (650 weight parts), which had been obtained by degradation of hydrogenated rapeseed oil (IV = 66), and glycerin (107 weight parts) were subjected to similar esterification and post-treatments as in Oil A to produce Oil C.

[0045]

#### (2) Analysis of constituent fatty acids in diglycerides

Diglyceride fractions in each oil were collected by a column chromatograph (manufactured by Wako Pure Chemical Industries, Ltd.; after triglycerides fractions were eliminated with "Wako Gel C-200" and hexane, the diglyceride fractions were obtained with hexane/diethyl ether (70/30). Following the procedures of "Adjustment Method of Fatty acid Methyl Esters" and "Composition of Fatty Acids" in "Standard Methods for the Analysis of Fats, Oils and Related Materials" compiled by The Japan Oil Chemists' Society, the diglyceride fractions were then analyzed by gas chromatography. From retention times and peak area ratios on a chart so obtained, the distribution of fatty acids in the diglycerides was determined. The analytical results are shown in Table 1.

[0046]

[Table 1]

|                         | Oils |      |      |
|-------------------------|------|------|------|
|                         | A    | B    | C    |
| TG                      | 13.2 | 11.6 | 14.0 |
| DG                      | 85.9 | 87.2 | 84.6 |
| 1,3DG                   | 59.3 | 60.2 | 58.3 |
| MG                      | 0.8  | 1.1  | 1.3  |
| FFA                     | 0.1  | 0.1  | 0.1  |
| Plant sterols (%)       | 0.3  | 0.3  | 0.2  |
| Citric acid (ppm)       | 5    | 5    | 5    |
| Constituent fatty acids |      |      |      |
| C16:0                   | 3.1  | 5.2  | 4.4  |
| C18:0                   | 1.3  | 2.1  | 16.3 |
| C18:1                   | 38.0 | 60.9 | 73.5 |
| C18:2                   | 47.9 | 20.3 | 2.0  |
| C18:3                   | 8.3  | 10.0 | 0.8  |

[0047]

Invention Products 1-5 & Comparative Products 1-3

To Oil A, Oil B and Oil C (each, 100 parts), vitamin E, vitamin C, citric acid and a plant sterol were added to produce Invention Products 1-5 and Comparative Products 1-3. A formula table is shown in Table 2.

[0048]

[Table 2]

| Ingredients (weight parts)                  | Invention Products |       |       |       |       |  | Comparative Products |     |       |
|---|--------------------|-------|-------|-------|-------|--|----------------------|-----|-------|
|   | 1                  | 2     | 3     | 4     | 5     |  | 1                    | 2   | 3     |
| Oil A                                       | 100                |       |       | 100   | 100   |  |                      | 100 | 100   |
| Oil B                                       |                    | 100   | 100   |       |       |  |                      |     |       |
| Oil C                                       |                    |       |       |       |       |  | 100                  |     |       |
| Citric acid <sup>1)</sup>                   | 0.002              | 0.002 | 0.002 | 0.002 | 0.003 |  | -                    | -   | -     |
| Glyceryl citrate stearate <sup>2)</sup>     | 0.4                | -     | -     | -     | -     |  | -                    | -   | -     |
| Glyceryl citrate oleate <sup>3)</sup>       | -                  | 0.25  | -     | -     | -     |  | -                    | -   | -     |
| Glyceryl succinate stearate <sup>4)</sup>   | -                  | -     | -     | 0.25  | 0.25  |  | -                    | -   | -     |
| Vitamin E <sup>5)</sup>                     | 0.1                | 0.15  | 0.15  | 0.2   | 0.2   |  | -                    | -   | 0.2   |
| Vitamin C derivative <sup>6)</sup>          | 0.015              | 0.05  | 0.05  | 0.025 | 0.025 |  | -                    | -   | 0.025 |
| Polyglyceryl fatty acid ester <sup>7)</sup> | -                  | -     | -     | 0.075 | 0.075 |  | -                    | -   | -     |
| Plant sterol <sup>8)</sup>                  | -                  | -     | -     | -     | 4.0   |  | -                    | -   | -     |

1) "Citric Acid" (product of ADM (Archer Daniels Midland Co.))

2) "Sun Soft 621 B" (product of TAIYO KAGAKU CO., LTD.)

3) "Poem K-37" (product of Riken Vitamin Co., Ltd.)

4) "Step SS" (product of Kao Corporation)

5) "E Oil 400" (product of Riken Vitamin Co., Ltd.)

6) "Vitamin C Palmitate" (product of Roche Vitamins Ltd.)

7) "THL-3" (product of Sakamoto Yakuhin Kogyo Co., Ltd.)

8) "Phytosterol S" (Tama Biochemical Co., Ltd.)

[0049]

## Example 2 High-humidity Storage Test

The oil compositions described in Table 2 were stored without stoppers in an environment of 40°C and 75% humidity. Their water contents were measured with time by the Karl Fisher method. When their water contents arose to 0.2%, the oil compositions were transferred into 100-mL sample bottles and the bottles were then tightly sealed. After they were allowed to stand at room temperature for 24 hours, their conditions were visually observed.

As a result, the oil compositions which contained the ingredients (A), (B), (C) and (D) as in the present invention did not develop turbidity even under the high-humidity conditions as shown in Table 3.

[0050]

[Table 3]

| Invention products |       |       |       |       | Comparative products |        |        |
|--------------------|-------|-------|-------|-------|----------------------|--------|--------|
| 1                  | 2     | 3     | 4     | 5     | 1                    | 2      | 3      |
| Clear              | Clear | Clear | Clear | Clear | Solidified           | Turbid | Turbid |

[0051]

## Example 3 Frying Test

Separately using the oils of 0.2% water content, frying was conducted by a method to be described below. Emission of an unpleasant odor during cooking and the flavors and textures

of the cooked foods were organoleptically ranked.

Using the oils prepared in Example 2, frying was conducted in the order of *tempura*, fried pork fillet and fried chicken under the following conditions.

Amount of oil: 600 g (Chinese frying pan)

Temperature of oil: 180°C, heated on a gas cooking stove  
(medium flame)

<Tempura>

|   |   |
|---|---|
| Frying ingredients: Shrimps (black tiger) | 8 |
| Lotus root (sliced)                       | 8 |
| Pumpkin (sliced)                          | 8 |
| Green peppers<br>(each cut in halves)     | 8 |
| Egg plants<br>(each cut in halves)        | 8 |

|                     |       |
|---------------------|-------|
| Batter: Wheat flour | 100 g |
| Eggs                | 50 g  |
| Water               | 150 g |

<Fried pork fillets>

Frying ingredients: Pork fillets (cut in 2 cm thick round  
slices)

Coating: The pork fillet slices were each coated with  
wheat flour, beaten eggs and bread crumbs in  
this order.

After cooking the *tempura* as described above, the oils  
were individually stored at room temperature for 1 week in oil

pots. To 300 g aliquots of the oils, the corresponding oils (300 g, each) shown in Table 2 were added, and fried pork fillets were cooked at 180°C.

<Fried chicken>

Frying ingredient: Chicken (dark meat) (bite-size)

Breading: The frying ingredient was breaded with a frying mix (product of Nisshin Flour Milling Co., Ltd.)

After cooking the fried pork fillets as described above, the oils were individually stored at room temperature for 1 week in oil pots. To 300 g aliquots of the oils, the corresponding oils (300 g, each) shown in Table 2 were added, and fried chicken was cooked at 180°C.

At the time of the completion of the frying, the edible oils were ranked based on the emission of an unpleasant odor and also in the flavors and textures of the fried foods by 10 panelists in accordance with the following ranking standards.

(Emission of unpleasant odor during cooking)

A: Extremely good without any unpleasant odor or deteriorated odor.

B: Good without a substantial unpleasant odor or deteriorated odor.

C: An unpleasant odor or deteriorated odor is felt a little.

D: An unpleasant odor or deteriorated odor is felt.

(Flavors and textures of fried foods)



- A: Extremely good without any unpleasant taste such as an acidic taste or bitter taste or substantial greasiness.
- B: Good without a substantial unpleasant taste such as an acidic taste or bitter taste or substantial greasiness.
- C: An unpleasant taste such as an acidic taste or bitter taste or greasiness is felt a little.
- D: An unpleasant taste such as an acidic taste or bitter taste or greasiness is felt, and the flavor is poor.

[0052]

As shown in Table 4, the resultant, fried foods were all good in flavor, external appearance and the like. Further, the smells of the edible oils during the frying were also good. The edible oils according to the present invention were successfully used for the edible oil applications.

[0053]

[Table 4]

|                   |  | Invention products |   |   |   |   | Comparative products |   |   |
|-------------------|--|--------------------|---|---|---|---|----------------------|---|---|
|                   |  | 1                  | 2 | 3 | 4 | 5 | 1                    | 2 | 3 |
| Tempura           | Emission of unpleasant odor during cooking | B                  | B | B | B | A | D                    | D | C |
|                   | Flavor and texture of cooked food          | A                  | A | B | A | A | D                    | D | C |
| Fried pork fillet | Emission of unpleasant odor during cooking | B                  | B | B | B | A | D                    | D | D |
|                   | Flavor and texture of cooked food          | B                  | A | B | A | A | D                    | D | C |
| Fried chicken     | Emission of unpleasant odor during cooking | B                  | B | B | B | B | D                    | D | D |
|                   | Flavor and texture of cooked food          | B                  | B | B | B | A | D                    | D | D |

[0054]

## Example 4 Low-Temperature Storage Test

The oil compositions of the formulas shown in Table 2 (Invention Products 3,4 and Comparative Products 1,3) were individually placed in glass-made sample bottles, and the bottles were tightly sealed. Subsequent to storage at 0°C for 24 hours, conditions of the oil compositions were visually observed.

[0055]

[Table 5]

| Invention products        |       | Comparative products |        |
|---------------------------|-------|----------------------|--------|
| 3                         | 4     | 1                    | 3      |
| Clear with some turbidity | Clear | Solidified           | Turbid |

[0056]

As a result, the oil compositions which contained the ingredients (A), (B), (C) and (D) as in the present invention did not develop turbidity even under the low-temperature storage conditions as shown in Table 5.

[0057]

## Example 5 Mayonnaise

(weight parts)

|                                  |      |
|----------------------------------|------|
| Oil (Invention Product)          | 65.0 |
| Egg yolk (phospholipase treated) | 15.0 |
| Vinegar (acidity: 10%)           | 7.0  |
| Sugar                            | 1.0  |
| Sodium glutamate                 | 0.4  |

|                         |      |
|-------------------------|------|
| Salt                    | 0.3  |
| Mustard (powder)        | 0.3  |
| Thickener (xanthan gum) | 0.1  |
| Water                   | 10.9 |

The materials other than the oil was agitated and mixed in a homomixer. The oil was then added dropwise, followed by pre-emulsification in the homomixer. The thus-obtained pre-emulsified mixture was homogenized in a colloid mill to produce mayonnaise (pH 4.0). The mayonnaise so obtained was good in flavor, external appearance, emulsion properties and the like. The oil composition according to the present invention was successfully used for the o/w emulsion application.

[0058]

#### Example 6 Spread

(Oil phase)

|                                    | (weight parts) |
|------------------------------------|----------------|
| Oil (Invention Product 4)          | 33.38          |
| Hydrogenated palm oil (IV = 2)     | 4              |
| Hydrogenated soybean oil (IV = 43) | 2              |
| Monoglycerides                     | 0.5            |
| Lecithin                           | 0.5            |
| Polyglyceryl ricinoleate           | 0.5            |
| Flavor                             | 0.1            |
| Vitamin E                          | 0.02           |

(Water phase)

(weight parts)

|                 |      |
|-----------------|------|
| Distilled water | 57.4 |
| Skimmilk powder | 0.3  |
| Salt            | 1.3  |

The oil phase and water phase were prepared, and were then mixed and emulsified by a homomixer. The thus-obtained emulsion was quickly chilled by conventional procedure to plasticize the same and hence, to prepare a spread. The spread so obtained was good in flavor, external appearance, emulsion properties and the like. The oil composition according to the present invention was successfully used for the w/o emulsion application.

[Advantageous Effects of the Invention]

According to the present invention, it is possible not only to markedly improve the problem of turbidity and solidification typical to diglycerides but also to pronouncedly prevent the occurrence of an unpleasant odor during cooking and hence, to substantially improve work efficiency by combining a specific organic acid, an antioxidant and a plant sterol with an oil or fat, which contains diglycerides having an effect to curtail the accumulation of body fat and to prevent adiposis.

[Document Name] ABSTRACT

[Abstract]

[Object] This invention relates to oil or fat compositions, each of which is high in the content of diglycerides having excellent health-promoting functions and even in cold temperature areas or high-humidity areas, is prohibited in the occurrence of solidification or turbidity and is good in work efficiency during cooking and also in the flavor and texture of cooked foods.

[Means for Achieving the Object] An oil or fat composition comprising the following ingredients (A), (B), (C) and (D):

(A) 100 weight parts of an oil or fat containing 60 to 100 wt% of diglycerides in which a content of unsaturated fatty acids in constituent fatty acids is from 80 to 100 wt%;

(B) 0.001 to 1 weight parts of a carboxylic acid selected from C<sub>2-8</sub> hydroxycarboxylic acids, dicarboxylic acids and tricarboxylic acids, and salts and derivatives thereof;

(C) 0.001 to 5 weight parts of an antioxidant; and

(D) 0.05 to 4.7 weight parts of a plant sterol.

[Selected Figure of Drawings] None.